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TECHNICAL REPORT 8207

TERTIARY TREATMENT OF EFFLUENT FROM HOLSTON AAP
INDUSTRIAL LIQUID WASTE TREATMENT FACILITY
I. BATCH CARBON ADSORPTION STUDIES:
TNT, RDX, HMX, TAX, AND SEX

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PREPARED FOR
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PREFACE

The research reported herein was performed at the request of Large Caliber Weapons Systems Laboratory, ARRADCOM, Dover, NJ, under R&D Project No. 1L162720D048, "Tertiary Treatment of Holston AAP Industrial Waste Treatment (ILWT) Effluent," Mr. Bossie Jackson, Jr., Energetic Systems Process Division, Project Officer. This study is part of the DARCOM Pollution Abatement and Environmental Control Technology Program conducted by the U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, MD. High performance liquid chromatography (HPLC) analyses were performed at USAMBRDL by Mr. Ernst E. Brueggemann, and statistical analysis of data was carried out by Mr. Paul H. Gibbs.



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INTRODUCTION

Holston Army Ammunition Plant (HSAAP) will produce 125 million gallons of wastewater per day at full mobilization. The current design of an MCA (Military Construction Army) project for the Industrial Liquid Waste Treatment Facility (ILWTF) at HSAAP does not provide a tertiary mode of treatment for the removal of pollutant chemicals which survive primary and secondary treatment. There is evidence that RDX, HMX, and TNT, as well as biproduct nitramines such as hexahydro-1(N)-acetyl-3,5-dinitro-1,3,5-triazine (TAX) and octahydro-1(N)-acetyl-3,5,7-trinitro-1, 3,5,7-tetrazocine (SEX) will survive secondary biological treatment, at least in part, and may adversely affect aquatic organisms in receiving waters. A problem is foreseen in complying with proposed ambient criteria of 0.06 mg/L for TNT and 0.3 mg/L for RDX/HMX for protection of aquatic life, based on studies by USAMBRDL.

This, the first in a series of studies in tertiary treatment technologies, concerns comparative removal efficiencies for the five HSAAP munitions production wastewater contaminants by granular activated carbon in the batch mode. Organic molecules are considered to be attracted to carbon surfaces by Van der Waals (nonbonded) forces. The equilibrium for formation of a monomolecular adsorption layer is described by the Langmuir equation:

$$q_e = \frac{Q^0 b C}{1 + b C}$$

where q_e is the amount of organic solute adsorbed per unit weight of carbon at concentration C , Q^0 is the amount of solute required per unit weight of carbon to completely saturate the adsorption capacity, and b is a constant related to the energy of adsorption.

For analysis of data from wastewater treatability studies, the Freundlich equation, an empirical relationship, is most commonly employed:

$$q_e = K C^{1/n}$$

where K and n are constants. The Langmuir and Freundlich equations are equilibrium expressions, but the viability of granular activated carbon adsorption as a wastewater treatment option depends not only on the adsorption equilibrium, but on the kinetics of adsorption. Carbon adsorption studies suitable for process design must employ continuous flow carbon columns and be tested with authentic wastewaters. The batch adsorption studies described in this report are intended to test the general feasibility of granular activated carbon treatment for removal of potentially toxic munitions wastewater constituents.

The previous work most relevant to this study was reported in 1976 by Layne and Tash,¹ who carried out batch adsorption tests with mixtures of RDX and TNT on granular activated carbon (GAC, Calgon F300, -200 mesh). They concluded that each compound interfered with adsorption of the other and that TNT was preferentially adsorbed, an exception to the general rule stated by Eckenfelder and Ford² that "a compound in a mixed solution is more readily adsorbed than it is when that compound is in the solution alone."

RESULTS AND DISCUSSION

Adsorption on granular activated carbon (Calgon F300, -200 mesh) was measured for each chemical over its maximum anticipated concentration, i.e., 20 to 25 mg/L or less (depending on its water solubility) down to the limit of detectability, which is 0.1 to 0.4 mg/L, depending on the compound and on the analytical procedure. Freundlich equations for each chemical, run individually, are listed below and presented graphically in Figure 1.

$$\text{TNT: } q_e = 0.3370 C^{1/5.429}$$

$$\text{RDX: } q_e = 0.1118 C^{1/2.938}$$

$$\text{HMX: } q_e = 0.1682 C^{1/2.169}$$

$$\text{TAX: } q_e = 0.1002 C^{1/3.498}$$

$$\text{SEX: } q_e = 0.2190 C^{1/2.853}$$

Log-log plots are generally linear at carbon-to-substrate ratios of 1:1 and higher. At lower ratios there is evidence for saturation of adsorption sites, except for RDX (see data from Table 4). At this stage the chemicals fall into three categories in terms of slopes: TNT, for which removal is least sensitive to concentration, RDX, HMX, and SEX, which are more dependent on concentration but not statistically separable from each other at the 95 percent level; and TAX, which is not classifiable (i.e., not distinguishable from TNT or the others). Statistical comparisons of slopes and intercepts are presented in Tables 1 and 2. In the same category as RDX, HMX, and SEX is adsorption of a mixture of TNT, RDX, TAX, SEX, and HMX (in the appropriate ratio of 25:24:20:6:5, respectively), arbitrarily reported in terms of mg/L "total nitro bodies."

Values derived for K and n are in satisfactory agreement with those (0.30 L/mg and 6.35, respectively) extrapolated from graphical data presented for TNT by Layne and Tash;¹ parameters extrapolated for RDX (0.066 L/mg and 1.90, respectively) indicate a somewhat greater concentration dependence for adsorption than was measured at USAMBRDL. However, both sets of results are in agreement in demonstrating the effectiveness of granular activated carbon in TNT removal; the flat slope of the Freundlich plot (Fig. 1) reflects a high level of adsorption efficiency, even at very low TNT concentrations. RDX, HMX, and SEX give significantly steeper slopes and are less efficiently removed, particularly at low concentrations (< 1 mg/L).

In terms of intercepts, the chemicals fall into three categories: TNT, which is most efficiently removed at all measured concentrations; HMX and SEX, which are less efficiently removed than TNT; and RDX and TAX, which are least efficiently removed. Apparently, substitution of a single acetyl group for a nitro group has no significant effect on adsorption for the nitramines. [Although relative adsorption efficiencies are commonly related inversely to water solubilities, it is not remarkable when such is not the case, as is evident here. Water solubilities are related to standard state (crystal lattice) energies, while adsorption efficiencies are related to Van der Waals forces, for which the dependence on molecular structure may be quite different.]

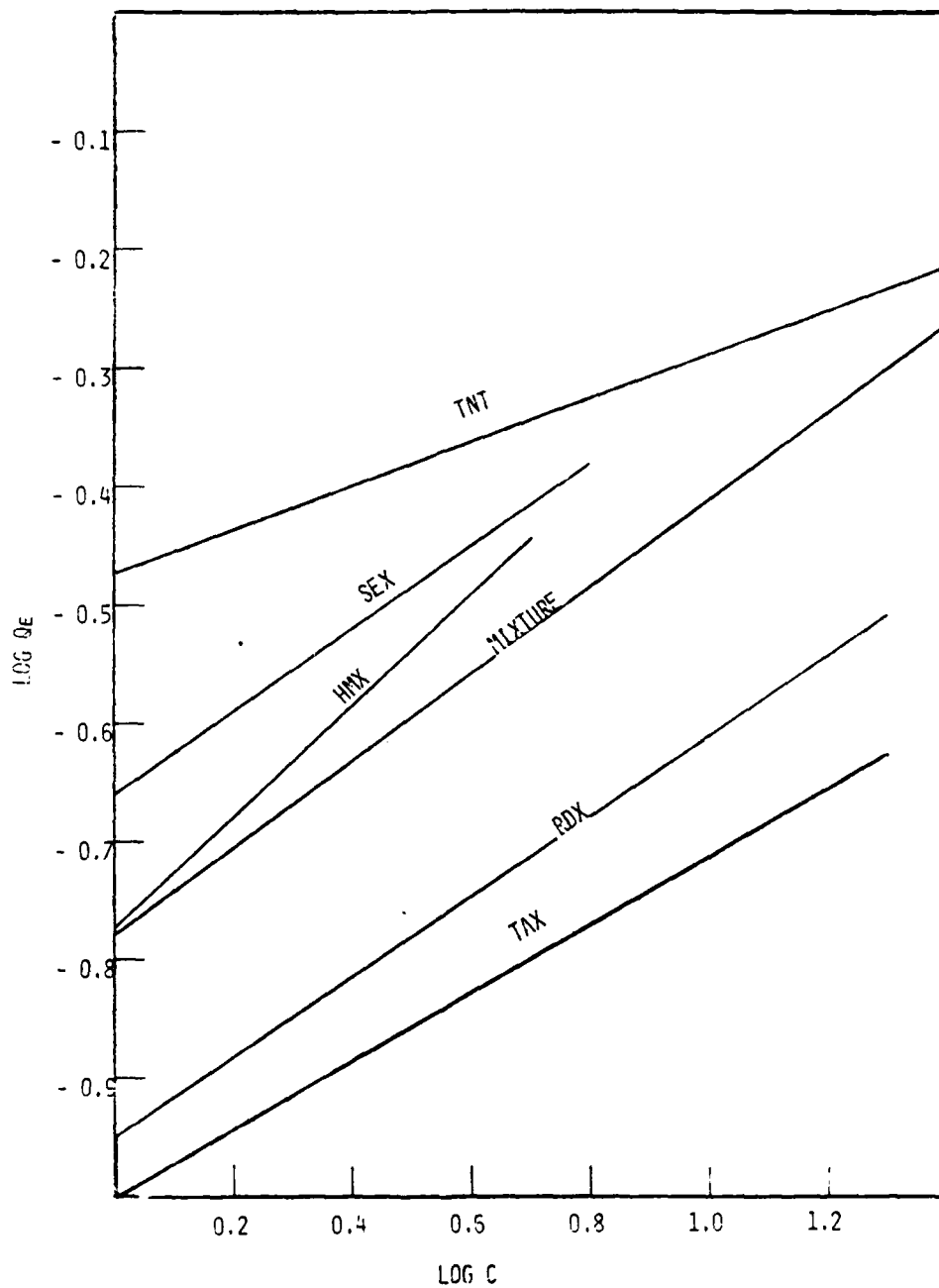


Figure 1. Regression lines for adsorption of chemicals on granular activated carbon.

TABLE 1. COMPARISON OF REGRESSION EQUATION SLOPES^a

	TNT	TAX	RDX	SEX	Mixture
HMX	p < 0.05 ^b	(NS) ^c	(NS)	(NS)	(NS)
Mixture	p < 0.05	p < 0.05	(NS)	(NS)	
SEX	p < 0.05	(NS)			
RDX	p < 0.05	p < 0.05			
TAX	(NS)				

a. Data from Table 7.

b. Intercepts differ at confidence level >95%.

c. Intercept difference not significant (p > 0.05).

TABLE 2. COMPARISON OF REGRESSION EQUATION INTERCEPTS^a

	HMX	TAX	RDX	SEX	Mixture
TNT	ND ^b	p < 0.05 ^c	ND	ND	ND
Mixture	(NS) ^d	ND	p < 0.05	p < 0.05	
SEX	(NS)	p < 0.05	p < 0.05		
RDX	p < 0.05	ND			
TAX	p < 0.05				

a. Data from Table 7.

b. Comparison not done.

c. Intercepts differ at confidence level >95%.

d. Intercept difference not significant (p > 0.05).

Total nitrobody adsorption in the mixture is significantly less efficient than adsorption of TNT and no more efficient than adsorption of HMX and SEX taken individually, in contrast to the general rule for mixtures but in agreement with the findings of Layne and Tash¹ for TNT and RDX. Log-log plots of Freundlich isotherms for RDX and HMX in the mixture (Fig. 2) are statistically parallel (95 percent confidence limits) in the linear portion to the same components examined individually, but have lesser intercepts, an indication of competition for adsorption sites. (The same is probably true for TNT, but only two points could be analyzed for the mixture.) The same plots for TAX and SEX in the mixture are not parallel to the corresponding lines for the individual components, and may not be linear. (See data from Table 5. If

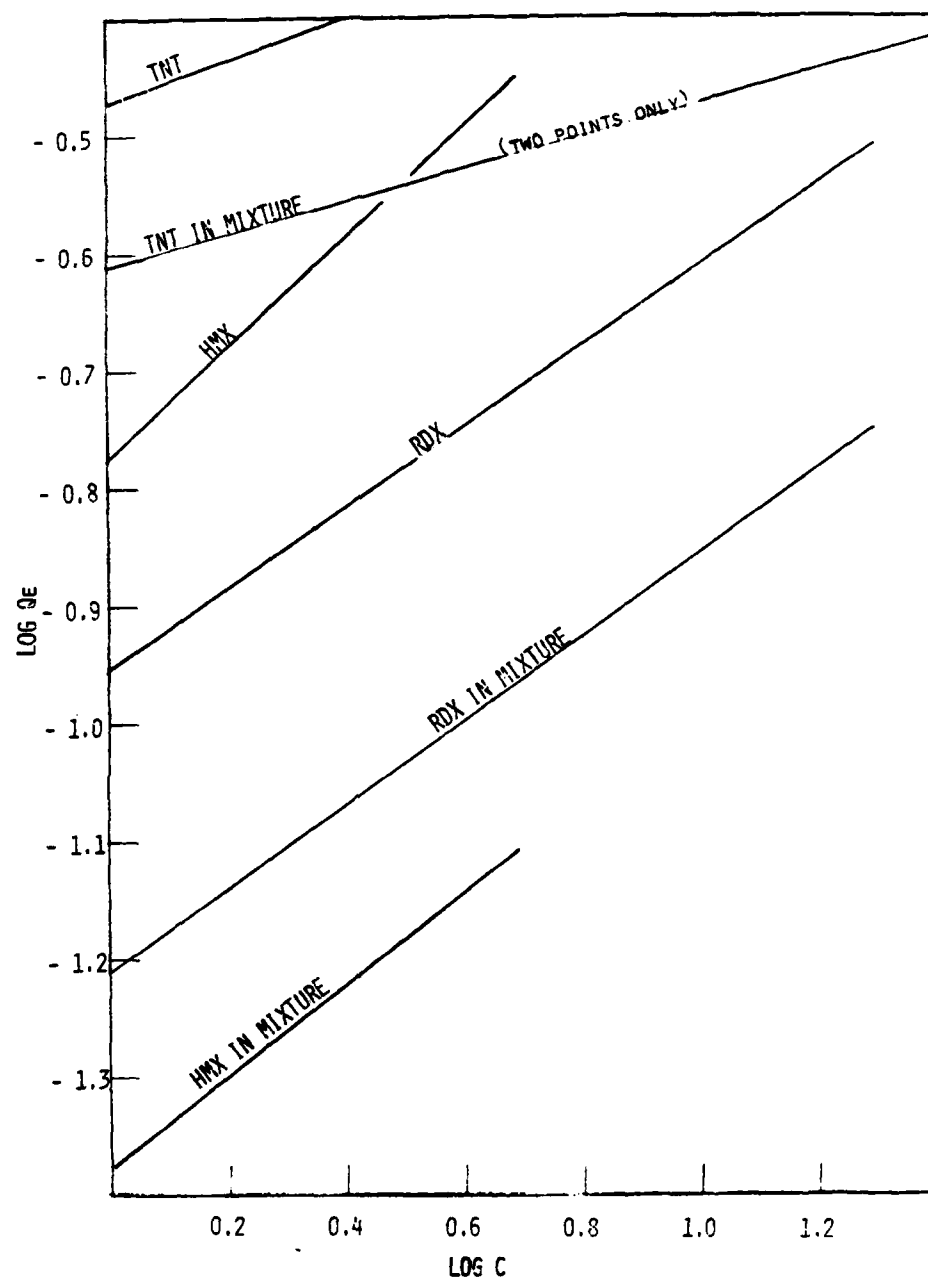


Figure 2. Regression lines for adsorption of chemicals from a mixture on granular activated carbon.

linear, these plots would have negative slopes, which are theoretically meaningless.) Nonlinearity is reported by Layne and Tash for mixtures of RDX and TNT, and some mixtures exhibit maxima in the Freundlich plots for RDX.

SUMMARY AND CONCLUSIONS

Although the results developed here show that each of the five constituents is readily removed from uncontaminated water by granular activated carbon when present alone, these results do not necessarily endorse the choice of GAC as appropriate tertiary treatment technology for nitramine-contaminated wastewaters. The decision to proceed with GAC treatment must depend on continuous tests using GAC columns and authentic or synthetic wastewaters containing nitramines and nitrobodyies, so that both kinetic and equilibrium effects can be evaluated. With respect to the latter the USAMBRDL results are not encouraging, since they indicate that the nitramines will be adsorbed in a series of bands (as in chromatography) at the tail end of the column and will be progressively displaced by TNT well before TNT achieves breakthrough. Layne and Tash argue similarly from their data that the use of carbon columns for removal of RDX/TNT mixtures is questionable, because RDX will be strongly desorbed as the column approaches breakthrough for TNT, making it possible for the effluent RDX concentration to exceed temporarily the influent concentration. This will have to be established through further research.

EXPERIMENTAL PROCEDURES

MATERIALS

1,2,3,4,5,6-Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) and 1,2,3,4,5,6,7,8-octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) were provided by HSAAP and were of washed, crude quality (Table 3). Both were air-dried to constant weight before use. 1-Acetyl-1,2,3,4,5,6-hexahydro-3,5-dinitro-1,3,5-triazine (TAX) and 1-acetyl-1,2,3,4,5,6,7,8-octahydro-3,5,7-trinitro-1,3,5,7-tetrazocine (SEX) were prepared by SRI International by methods described previously,^{3,4} and were used as received (Table 3). 2,4,6-Trinitrotoluene (TNT) was synthesized at USAMBRDL and recrystallized from ethanol. Granular activated carbon (GAC; Calgon F300) was pulverized and screened to -200 mesh. A single batch was used for all experiments.

TABLE 3. CONSTITUENTS OF TEST SUBSTANCES

Substance	Purity, % (dry basis)	Other Constituents, %
TNT	est. >99	
RDX	88.61	HMX, 11.39
HMX	98.76	RDX, 1.24
TAX	99	
SEX	96.7	HMX 2.4, DADN ^a 0.9

a. 1,5-Diacetyloctahydro-3,7-dinitro-1,3,5,7-tetrazocine.

PROCEDURE

To each of six 600 mL beakers were added a weighed quantity of powdered carbon and 500 mL of a solution of the munitions constituent or constituents in deionized water. The mixtures were stirred at 100 rpm in a Phipps and Bird, Inc. jar test apparatus for 24 hours, then allowed to settle for approximately 1 hr before analysis. [The mixture of constituents was prepared by dissolving TNT (87.71 mg), RDX (84.44 mg), HMX (17.05 mg), TAX (69.70 mg), and SEX (20.95 mg) in 3.5 L of deionized water to give a solution containing approximately 80 mg/L total nitrobodyes.] Adsorption studies were usually run from noon to noon at ambient temperature, which was 22° to 24°C at midday but occasionally dropped to 18° C at night. The initial pH value of the deionized water ranged from 5 to 5.5 and did not change significantly in the course of the adsorption test. Adsorption data are presented in Tables 4 and 5.

ANALYSIS BY DIRECT UV MEASUREMENT

For each of the five constituents, dissolved in deionized water, optical absorbance vs. concentration curves were prepared (based on λ_{\max}) using the Beckman 5230 UV spectrophotometer, 1 cm quartz cells, and a scan speed of 4 nm/sec. Because Beer's law was not generally observed above 5 mg/L, all concentration values reported in Table 4 are extrapolated from these curves. (See Table 6 for wavelength/intensity data.) For the mixture, containing approximately 80 mg/L total nitrobodyes, the actual nitrobody concentration was determined by diluting to 1/4 initial concentration (at which level the absorbance vs. concentration curves are nearly linear for each component) and then, based on the known ratios of components, finding the individual values of absorbance which combine to give the observed absorbance for the mixture. For direct UV analysis, carbon adsorption samples were centrifuged to remove suspended carbon. (Strong absorption interference in the 230 nm region was leached from either of two grades of Whatman filter paper initially used to remove carbon.) Individual components of the mixture were analyzed by HPLC as described below.

TABLE 4. ADSORPTION OF NITRAMINES/NITROBODIES
ON CARBON (DIRECT UV ANALYSIS)

Chemical	Test Run-Sample	Concentration mg/L	Carbon mg/L	q_e^a
TNT	4-0	25.3		
	4-1	19.6	12.00	0.5 ^b
	4-2	12.3	25.78	0.304
	4-3	2.7	51.98	0.435
	4-4	0.4	77.86	0.320
	4-5	0.2	122.20	0.205
	4-6	<0.1	250.78	
	5-0	50.8		
	5-1	37.4	26.04	0.515 ^b
	5-2	22.8	50.40	0.556
	5-3	3.7	100.50	0.469
	5-4	0.7	150.14	0.334
RDX	4-0	21.0		
	4-1	14.6	22.40	0.286
	4-2	10.5	43.38	0.242
	4-3	3.3	102.26	0.173
	4-4	0.8	196.88	0.103
	4-5	0.1	408.0	0.051
	4-6	7.8	60.06	0.220
HMX	6-0	5.20		
	6-1	3.49	5.86	0.292
	6-2	2.86	11.64	0.201
	6-3	0.93	18.60	0.230
	6-4	0.30	33.22	0.148
	6-5	0.10	62.60	0.080
	6-6	0.07	125.20	0.041
TAX	2-0	21.7		
	2-1	18.8	20.94	0.138 ^b
	2-2	10.6	50.42	0.185
	2-3	1.5	198.76	0.102
	3-0	17.2		
	3-1	13.4	21.40	0.180 ^b
	3-2	9.2	41.00	0.194
	3-3	6.3	61.08	0.178
	3-4	2.8	100.60	0.143
	3-5	0.45	203.60	0.082
	3-6	<0.1	398.24	
SEX	1-0	5.16		
	1-1	3.60	5.14	0.304
	1-2	1.25	16.02	0.244
	1-3	0.10	51.54	0.098
	2-0	6.00		
	2-1	4.17	6.64	0.276
	2-2	2.20	13.20	0.288
	2-3	1.25	18.44	0.258
	2-4	0.35	31.22	0.191
	2-5	0.15	61.46	0.095
Mixture	1-0	71.0		
	1-1	48.8	40.0	0.542 ^b
	1-2	27.3	70.4	0.550
	1-3	10.4	161.4	0.375
	1-4	3.8	225.5	0.298
	1-5	1.1	404.0	0.173
	1-6	0.2	798.0	0.090

a. q_e = Chemical removed (mg/L) ÷ carbon applied (mg/L).

b. Point not used in calculation of regression line.

TABLE 5. ADSORPTION OF MIXED NITRAMINES/NITROBODIES
ON CARBON (HPLC ANALYSIS)

Test Run-Sample	Carbon mg/L	TNT mg/L	RDX mg/L	HMX mg/L	TAX mg/L	SEX mg/L
1-0		23.48	22.60 ^a	4.71	26.16	5.85
1-1	40.9	9.87	15.42	2.37	25.99 ^b	5.06
1-2	79.4	2.06	11.63	1.16	21.70	3.69
1-3	161.4	<0.281 ^c	6.23	0.33	13.20	0.52
1-4	225.5		2.25	<0.234 ^c	4.97	<0.377 ^c
1-5	404.0		0.78		1.24	
1-6	798.0		<0.264 ^c		<0.318 ^c	

a. Calculated value.

b. Value not included in calculation of q_e .

c. Detection limit for HPLC method.

TABLE 6. UV ABSORBANCE DATA FOR MUNITIONS CHEMICALS

Chemical	λ_{max} , nm	Absorbance, %, at 5 mg/L	Minimum Readable Concentration, mg/L ^a
TNT	230±1	40.5±1	0.1
RDX	233±1	25.5±1	0.1
HMX	226±	33.5±1	0.1
TAX	233±1	16.5±1	0.1
SEX	222±1	29.0±1	0.1
Mixture	230±2	32.0±1	0.25

a. Estimated maximum error ± 0.05 , from standard concentration
vs. absorbance data.

ANALYSIS BY HPLC

The major portion of each sample was filtered through Whatman 2V fluted paper. The filtered sample was collected on a Sep-Pak^R C₁₈ cartridge as follows: the Sep-Pak was preconditioned by passing through, in sequence, 5 mL of methanol, 5 to 10 mL of deionized water, and 10 mL of air; then a 50 mL sample was passed through the Sep-Pak at a rate of 5 to 10 mL/min, followed by 5 mL of air, using a 50 mL syringe. The analytical procedure was adapted from Stidham (1979)⁵. Material to be analyzed was eluted from the Sep-Pak by passing through 4 mL of a 1:1 acetonitrile:water solution, followed by 10 mL of air. The eluent was centrifuged at 2000 rpm for 15 min before analysis. The Water HPLC with Model 720 systems controller, Model 730 data module, M450 detector, Model U6K injector, and Model M600A pumps was used according to the following operating parameters:

Column: RAD-PAK-A C₁₈

Mobile Phase: Pump A, 25% methanol in water
Pump B, 80% methanol in water

Gradient: 5-50% B in 25 min

Flow Rate: 1.7 mL/min

Detector Wavelength: 240 nm

Injection Volume: 20 μ L

Peak areas were compared with a standard curve prepared from solutions of 0.2, 0.4, 1.0, and 2.0 mg/L for each chemical, matched against 1,3-dinitrobenzene as internal standard. (Eluent samples were further diluted as required.)

Treatment of Data - Calculated values of q_e for each adsorption experiment are listed in Tables 4 and 5. These data were treated by linear regression; regression equations are presented in Tables 7 and 8 for the individual compounds and for the individual compounds in mixture, respectively. [By inspection, it was apparent that saturation of adsorption sites occurred for some compounds at low carbon-substrate ratios (generally less than 1:1), i.e., q_e was no greater than for higher ratios. These data points were omitted from the regression analysis.] Regression lines were compared by analysis of covariance according to the method of Brownlee (1965)⁶, with 95% confidence limits. Each compound, tested alone, was compared with every other compound and with the collective mixture with respect to slope (Table 1) and intercept (Table 2); and each compound, tested alone, was compared with the same compound in the mixture.

Sep-Pak is a registered trademark of Waters Associates, Inc., Milford, MA.

TABLE 7. REGRESSION EQUATIONS FOR GAC ADSORPTION DATA
(DIRECT UV ANALYSIS)

$\text{Log } q_e = \text{Log } K' + 1/n (\text{Log } C - \overline{\text{Log } C})^a$			
Chemical	$\text{Log } K' \pm \text{S.E.}$	$1/n \pm \text{S.E.}$	$\overline{\text{Log } C}$
TNT	-0.4146 ± 0.0227	0.1842 ± 0.0292	0.3137
RDX	-0.8098 ± 0.0055	0.3404 ± 0.0064	0.4165
HMX	-0.8848 ± 0.0673	0.4641 ± 0.1016	-0.2386
TAX	-0.8528 ± 0.0183	0.2859 ± 0.0260	0.5122
SEX	-0.7189 ± 0.0222	0.3505 ± 0.0384	-0.1696
Mixture	-0.6048 ± 0.0148	0.3680 ± 0.0167	0.4751

- a. $\overline{\text{Log } C}$, the average value of $\text{Log } C$, is introduced into the regression equation so that the regression lines in Figure 1 are centered about the average. For the Freundlich equations,

$$\text{Log } K = \text{Log } K' - 1/n \overline{\text{Log } C}$$

(The listed S.E. for $\text{Log } K'$ is actually the S.E. for $\text{Log } K$)

TABLE 8. REGRESSION EQUATIONS FOR GAC ADSORPTION DATA
FROM MIXTURE (HPLC ANALYSIS)

$$\text{Log } q_e = \text{Log } K' + 1/n (\text{Log } C - \overline{\text{Log } C})^a$$

Chemical	Log $K' \pm \text{S.E.}$	$1/n \pm \text{S.E.}$	$\overline{\text{Log } C}$
TNT ^b	-0.523	0.1338	0.654
RDX	-1.0024 \pm 0.0362	0.3181 \pm 0.0444	0.655
HMX	-1.384 \pm 0.0047	0.3899 \pm 0.0133	-0.014
TAX	-1.177 \pm 0.118	-0.0538 \pm 0.1258	0.812
SEX	-1.590 \pm 0.065	-0.1931 \pm 0.1189	0.329

- a. $\overline{\text{Log } C}$, the average value of Log C, is introduced into the regression equation so that the regression lines in Figure 2 are centered about the average. For the Freundlich equations,

$$\text{Log } K = \text{Log } K' - 1/n \overline{\text{Log } C}$$

(The listed S.E. for Log K' is actually the S.E. for Log K).

- b. Two points only.

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